Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

catena-Poly[[[bis(1*H*-imidazole-κN^3)zinc(II)]-μ-1,4-phenylenedioxydiacetato-κ^2*O*:*O*′] tetrahydrate]

Shan Gao,[a] Ji-Wei Liu,[a] Li-Hua Huo,[a] Hui Zhao[a] and Seik Weng Ng[b]*

[a]College of Chemistry and Chemical Technology, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, Kuala Lumpur 50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Received 2 August 2004
Accepted 23 August 2004
Online 28 August 2004

The zinc atom in the title compound, $\{[Zn(C_{10}H_8O_6)(C_3H_4N_2)_2]\cdot 4H_2O\}_n$, shows tetrahedral coordination; this atom, which lies on a position of site symmetry 2, is covalently bonded to the carboxyl O atom of one arm of the phenylenedioxydiacetate unit and datively bonded to the imidazole heterocycle. The polymeric chains propagate along the shortest axis of the orthorhombic unit cell and are assembled into layers. The phenylenedicarboxylate moiety lies on an inversion center and two of the three independent uncoordinated water molecules lie on twofold axes.

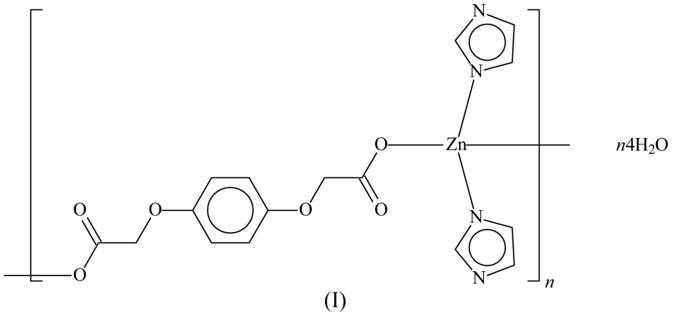
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.203
Data-to-parameter ratio = 17.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We will report elsewhere the structure of polymeric (1,4-phenylenedioxydiacetato)zinc(II) (Gao *et al.*, 2004); this compound did not react with benzimidazole under hydrothermal conditions. However, under different reaction conditions, the smaller five-membered ring analog, imidazole, afforded the title complex, (I), which crystallizes as a tetrahydrate (Fig. 1).



(I)

The phenylenedioxydiacetate dianion, which lies on an inversion center, links adjacent Zn atoms into a linear chain running along the shortest axis of the unit cell. The Zn atom, lying on a twofold rotation axis, is also coordinated by two heterocyclic ligands in a tetrahedral environment; the chains are assembled into a layered structure. Two of the three independent solvent water molecules lie on twofold axes. A tetrahedral geometry appears to be common for zinc dicarboxylate complexes of imidazole, as noted from a cursory search through the Cambridge Structural Database (Version 5.25; Allen, 2002). The Zn atom in the oxydiacetate (Baggio *et al.*, 1999), phthalate (Liu *et al.*, 2002; Baca *et al.*, 2003), isophthalate and terephthalate (Yang *et al.*, 2002) complexes adopt this geometry; these compounds, as well as the title compound, feature hydrogen-bonding interactions that consolidate the structures.

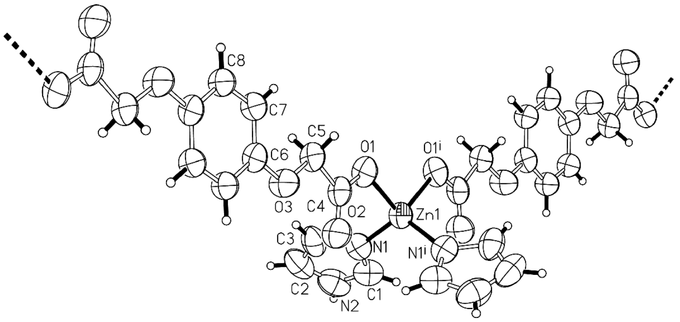


Figure 1
*ORTEP*II (Johnson, 1976) plot depicting a fragment of the polymeric chain structure of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. The solvent water molecules are not shown. [Symmetry code: (i) $\frac{5}{4} - x, \frac{5}{4} - y, z$.]

Experimental

Zinc diacetate dihydrate (4.38 g, 20 mmol) and imidazole (1.36 g, 20 mmol) were added to 1,4-phenylenedioxydiacetic acid (4.52 g, 20 mmol) in water. The pH of the mixture was adjusted to about 6 by the addition of 0.1 *M* sodium hydroxide. The solution was filtered. Colorless crystals separated from the filtrate after several days. Analysis calculated for $C_{16}H_{24}N_4O_{10}Zn$: C 38.61, H 4.86, N 11.26%; found: C 38.51, H 4.90, N 11.21%. IR (KBr pellet): 3442 (water), 1617 and 1429 (CO_2), 1232 and 1068 (C—O—C) cm^{-1}. The presence of four solvent water molecules in the formula unit was verified in the thermogram when a sample of the compound was heated from room temperature to 430 K (percentage loss: found 12.97%, calculated 12.86%). The final residual weight 16.43% (calculated 16.35%) corresponded to zinc oxide.

Crystal data

$[Zn(C_{10}H_8O_6)(C_3H_4N_2)_2]\cdot 4H_2O$
$M_r = 497.76$
Orthorhombic, *Fddd*
$a = 11.323$ (1) Å
$b = 18.517$ (2) Å
$c = 41.438$ (4) Å
$V = 8688$ (1) Å^3
$Z = 16$
$D_x = 1.522$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 22 377 reflections
$\theta = 3.2$–27.3°
$\mu = 1.19$ mm^{-1}
$T = 295$ (2) K
Prism, colorless
0.39 × 0.27 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.608$, $T_{\max} = 0.814$
19 839 measured reflections

2493 independent reflections
2045 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 27.5°$
$h = -14 \rightarrow 14$
$k = -24 \rightarrow 23$
$l = -53 \rightarrow 53$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.203$
$S = 1.09$
2493 reflections
142 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1307P)^2 + 17.4198P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.98$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	1.977 (3)	Zn1—N1	1.989 (4)
O1—Zn1—O1$^{\rm i}$	98.9 (2)	O1—Zn1—N1$^{\rm i}$	109.6 (1)
O1—Zn1—N1	107.3 (1)	N1—Zn1—N1$^{\rm i}$	121.7 (2)

Symmetry code: (i) $\frac{5}{4} - x, \frac{5}{4} - y, z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*n*···O1*w*	0.86	1.98	2.808 (8)	161
O1*w*—H1*w*1···O2$^{\rm ii}$	0.86	2.21	2.980 (8)	148
O1*w*—H1*w*2···O2*w*	0.89	2.40	3.12 (1)	139
O3*w*—H3*w*···O3	0.87	2.31	3.13 (1)	158
O2*w*—H2*w*···O1$^{\rm iii}$	0.86	2.12	2.889 (7)	148

Symmetry codes: (ii) $\frac{5}{4} - x, y, \frac{5}{4} - z$; (iii) $\frac{3}{2} - x, y - \frac{1}{4}, \frac{1}{4} + z$.

Two of the three crystallographically independent water molecules (O2*w* and O3*w*) lie on special positions at the Wyckoff 16*g* sites. The water molecules are probably disordered as there is enough empty space around them; however, the disorder could not be modeled. The extent of elongation of the displacement ellipsoids was reduced by restraining the parameters to approximate isotropic behavior. The H atoms of these molecules were placed at chemically sensible positions and their $U_{\rm iso}$ values set equal to $1.2U_{\rm eq}$ of the parent O atoms. The remaining H atoms were placed in calculated positions [aromatic C—H = 0.93 Å, aliphatic C—H = 0.97 Å and N—H = 0.86 Å; $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C,N})$], and were included in the refinement in the riding-model approximation.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); structure solution: *SHELXS*97 (Sheldrick, 1997); structure refinement: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

We thank the National Natural Science Foundation of China (No. 20101003), the Educational Committee Foundation of Heilongjiang Province, Heilongjiang University and the University of Malaya for supporting this study.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Baca, S. G., Filippova, I. G., Gerbeleu, N. V., Simonov, Y. A., Gdaniec, M., Timco, G. A., Gherco, O. A. & Malaestean, Y. L. (2003). *Inorg. Chim. Acta*, **344**, 109–116.

Baggio, R., Garland, M. T., Perec, M. & Vega, D. (1999). *Inorg. Chim. Acta*, **284**, 49–54.

Gao, S., Liu, J.-W., Huo, Li.-H., Zhao, H. & Ng, S. W. (2004). *Acta Cryst.* E**60**. Submitted.

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Liu, Q.-Y., Liu, P., Wang, J. & Chen, Y. (2002). *Chin. J. Struct. Chem.* **21**, 509–512.

Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2002). *CrystalStructure.* Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381, USA.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Yang, J.-H., Zheng, S.-L., Tao, J., Liu, G.-F. & Chen, X. M. (2002). *Aust. J. Chem.* **55**, 741–744.

m1330 Shan Gao *et al.* • $[Zn(C_{10}H_8O_6)(C_3H_4N_2)_2]\cdot 4H_2O$ *Acta Cryst.* (2004). E**60**, m1329–m1330